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Processing of ochre from Daitari Iron ore mines, Singbhum Craton, Eastern India for optimum utilisation

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Ochre, a naturally occurring powdery material, has been commonly appeared associated with different iron ore morpho-types in many iron ore deposits of the Singhbhum Craton, eastern India. It appears in yellow, red, gray and black colours. The present paper has described the characteristics of ochre occurring in the Daitari Iron Ore Mines in Odisha, India, and has discussed its processing for sustainable iron making and optimum utilization. Usually, ochre shows poor iron value, fine size and therefore being considered as a waste. However, in the present set-up, the ochre sample has shown ~60% Fe, 4% combined Al_2O_3 + SiO_2 and ~10% LOI, contributed by hematite, goethite and limonite phases. Though compositionally it appears to be a good candidate for iron making, it has remained unsuitable as blast furnace feed due to its fine particle size and high LOI content.

Attempts have been made to convert this powdery ochre to a lumpy form by adopting the pelletization technique. The sample below 150 µm size has been mixed thoroughly with three different charge mixes (bentonite, limestone and coke fines) and pellets have been prepared in a laboratory-scale disc pelletizer., Three sets of samples have been prepared with variable bentonite contents (0.5, 0.75 and 1%) keeping fixed limestone and coke amount. The green pellets from the pelletizer have been exposed to drop test and Green Compressive Strength (GCS), followed by thermal indurations at 1100, 1200 and 1300 °C. The porosity (%) and Cold Crushing Strength (CCS) (kg/pellet) of the indurated pellets have shown variations at three different temperatures. Pellets prepared with 1% bentonite content and indurated at 1300 °C have given 22% porosity and 255 kg/pellet CCS. XRD, optical and electron microscopy studies have revealed that recrystallization of hematite during induration has enhanced pellet strength and properties, while fayalite and calcium ferrite formation has provided stability through slag bonding. Pellets indurated at 1300 °C have shown Fe enrichment from ~60% to ~67% with ~3.2% combined Al_2O_3 + SiO_2, making them suitable as blast furnace feed. If this technique is adopted, an appreciable quantity of ochrous waste accumulated at mine sites can be converted to wealth.

Keywords: Daitari mine, Iron ore deposits, Ochre, Pelletization, Singhbhum Craton

1 Introduction

The study of ochre, one of the lithological units in the Banded Iron Formation of the Singhbhum Craton in Eastern India, has received little attention due to its low iron content and powdery nature Acharya[1]. Often it is referred to as ochrous shale because of its shaly appearance. Ochre can be found in various colours such as: yellow, red, grey, and black, within the mine profile of iron-rich states in India. As per their colour, these are termed as red ochre, yellow ochre, green earth, sienna, umber etc. The total reserves and resources of ochre have been estimated as 167.79 MT (NMI data based on the UNFC system, 2015) IBM[2]. Of these, approximately 36.93 MT are classified as 'Reserves,' while 130.86 MT fall under the 'Remaining Resources' category IBM[2]. Out of total resources, 87% are of red ochre, 11% are of yellow ochre and the remaining 2% are yet to be worked out IBM[3], IBM[4]. The yellow and red colour is due to presence of hydrous and anhydrous iron oxide respectively. The staining and colouring agents are purely of inorganic constituents. The amount of Fe in ochre varies between 20 and 70%.

Although an appreciable quantity of ochre is reported from various iron-rich states of India, as briefed below, it is usually considered as waste leading to disposal and environmental problems. The Government of India in Feb. 2015 notified 'Ochre' as a 'Minor Mineral.' Since then concerted efforts are being made to find out the suitable or beneficial use of ochre IBM[2].

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Occurrence of ochre is reported from different parts of India. The data published by Indian Bureau of Mines indicates that Rajasthan contributes 46% of total ochre deposit followed by Madhya Pradesh and Chhatishgarh having a share of 25% to total Indian distribution The remaining occurrences are reported from Gujrat, Maharastra. Andhra Pradesh, Telengana, Jharkhand, Tamil Nadu. Minor occurrences are reported from Himachal Pradesh, Haryana, Punjab, Uttar Pradesh, Arunachal Pradesh and Kerala etc. IBM[2], Krishnaswamy[5].

Channabasappa[6] located pockets and lenses of yellow ochre near the Masur area in Dharwar district resulting from the continuous weathering of hematite-phyllites and hematite-rich ferruginous quartzite. Gopal[7] reported around 9,00 tons reserve of both red and yellow ochre in Tiruchirapalli district, Tamil Nadu, which is primarily used for manufacturing paints. Srivastava[8] reported lenticular bands of red and yellow ochre (3 to 5' thick), within lateritised sandstones in the Jaitwara area of Satna dist., MP, with 90000 and 56000 tons reserve respectively, having 30% and 56% iron oxide. Middlemiss[9] documented ochre deposits in Jammu and Kashmir. Rao[10] reported red and yellow ochre in the northeast hills of Midnapur district, West Bengal containing thin calcareous and quartz veins with mica particles derived from schistose rock. Iyengar[11] noted the existence of inferior-quality red ochre in the form of soft gravel or lateritic soil on the hills of North Kanara district, Karnataka. Atram[12] observed the presence of an ochreous clay bed of inferior quality around the Sarsela region of MP. Biswas[13] identified yellow, pink, and mixed varieties of ochre, with Fe_2O_3 content ranging from 3.20% - 14.40%, located in Midnapur district in West Bengal, with a reserve estimated at 0.195 million tonnes. Misra[14] recorded the close association of red and yellow ochre with porous lithomarge around Varanasi, UP, which is primarily used for pottery and related wares. Cavallo[15] and Pandit explored the geology and petrography of ochre deposits from Rajasthan, suggesting that biotite alteration as the main factor responsible for the formationof yellow ochre.

Many researchers have documented occurrence of ochre throughout the country, yet its use is very much limited. Today, ochre is primarily used for colouring earthenware, household utensils, distempers, oil paints, lacquers, and primers and forimparting colour to paper and cement IBM[3], IBM[4]. Despite these wide range of applications, ochre is not utilized in large quantities, resulting stacking at mine sites that creates both environmental and disposal problems. A more effective and extensive use of ochre could be in the iron and steel industries as a feed stock for iron production, provided its composition is similar to that of iron ore. This shift could contribute to the country's goal of achieving a crude steel capacity of 300 MT by 2030 IBM[2].

In this contribution, we undertake a detailed study on the processing of red and yellow ochre that occur together in Daitari iron ore mine for its effective use in iron and steel industries. Our objectives were: (i) to characterize the mineralogical, chemical properties and thermal behavior of ochre from Daitari iron ore mines, Odisha; (ii) to pelletize the powdery material to a lumpy form for its use as blast furnace feed; (iii) to optimize the pelletization process by adjusting factors such as charge mix, induration temperature and time to achieve desired pellet properties like compressive strength and porosity.

2 Materials and Methods

2.1 Materials

Occurrence of several patches of ochre is seen at the North West side of the Daitari mines, Odisha. In one of the mine profiles (Fig. 1(a& b), both red and yellow ochre occur in close association. The chemical composition between these two does not show much variation. Hence, bulk representative groove samples (around 50kg) of ochre were collected from the said mine profile for laboratory investigation.

2.2 Methods

2.2.1 Characterization techniques

The particle size distribution of ochre samples is given in Table 1. As can be seen from the table by and large it is fine in nature and around 78% of the sample is below 150 µm. However, to bring it to a uniform size, the >150 µm samples were ground below 150 µm using a laboratory pulverizer and proper sieving. Around one gram of sample was subjected to XRD analysis (Malvern Panalytical, Almelo, Netherlands) to identify the mineral phases operating with Cu-Kα radiation at 40 kV and 30 mA with a scan rate of 2^o per minute. Thin polished sections of ochre sample were prepared to study the microstructure and identify the mineral phases using a reflected light microscope (DM4 P, Leica Microsystems Manufacturers, Germany) and a Scanning Electron

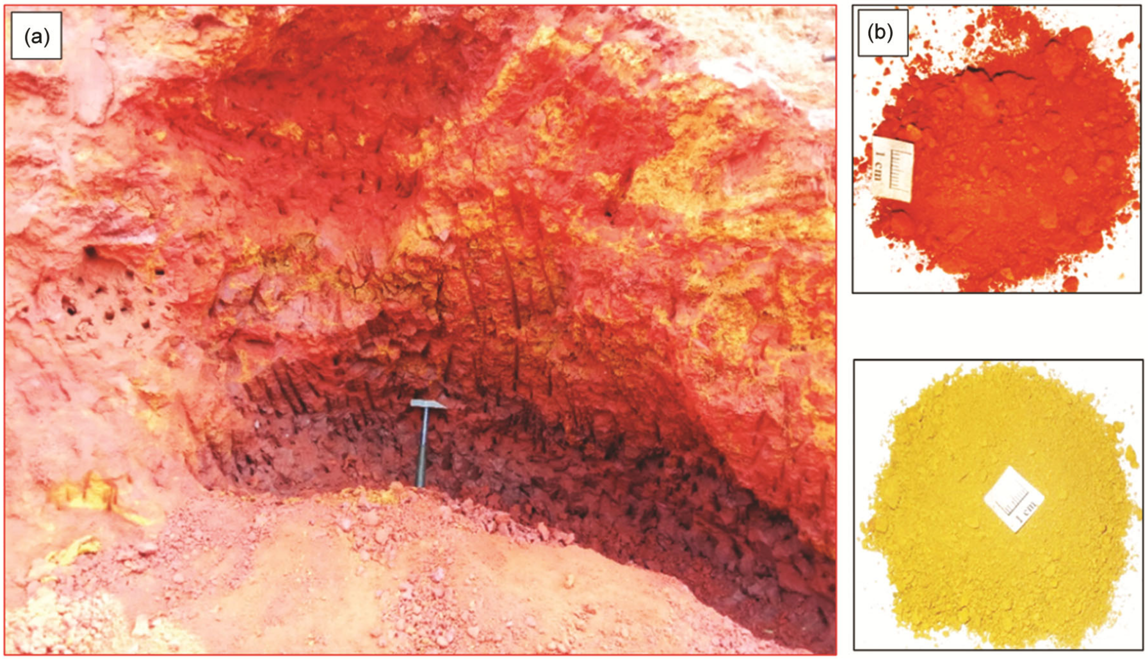

Fig. 1 — a) Field view of red and yellow ochre, & b) Megascopic view of red and yellow ochre.

Table 1 — Particle size distribution of ochre sample (in wt%)

Size, µm	-10+5.6	-5.6+2	-2+1	-1+150	-150+106	-106+75	-75+45	-45+2.5	-25
Ochre	3.1	4.7	4.2	10.7	7.3	11.1	15.6	7.5	35.8

microscope (ZEISS EVO 18). The chemical analysis of ochre samples was determined by X-ray fluorescence technique using a PAN analytical instrument (Model-Zetium 4.0 kW) in conjunction with the WROXI standard. Composition of different charge mix such as limestone and bentonite were determined by wet chemical analysis, while the composition of coke fines was found out by proximate analysis using a Leco Analyser.

2.2.2 Pelletization technique

Pelletization technique is generally used to convert either mine fines or beneficiated products to a lumpy form of desired specifications. It is favoured over other two agglomeration techniques such as sintering and briquetting due to its efficiency and environmental benefits. The pelletization process produce uniform, high quality pellets and requires less energy consumption.

Pellets are the balls of 8 to16 mm size produced from iron ores fines (< 150 µm) of different mineralogical and chemical composition. It is an oxidation process where the iron minerals convert to hematite or recrystallized hematite involving agglomeration of fine particles through heat treatment technique. The process of pelletization consists mainly of two steps: first green pellet and second indurate pellets Barik[16]. The mechanism and process of green pellet formation have been studied by many researcher Barik[16-18], Prusti[17-21], Patra[22].

2.2.3 The Pelletizer

The pelletization process was performed in a laboratory scale disc pelletizers shown in Fig. 2. The pellet mix was given manually to the disc pelletizer and appropriate amount of water was added time to time to maintain the moisture content of pellets. Green pellets of size ranging between 10-13 mm were separated manually from the pelletizer. About 15 numbers of green pellets from each batch were dropped from a height of 45 cm to a mild steel plate of one cm thick. The number of drops required to break the green pellets was noted. The average of drop number of ten pellets was taken as the drop number of that particular batch Nayak[23].

The Green Compressive Strength (GCS) of the green pellets was calculated using a universal tensile testing system (Shanta Engineering, Kolkata). The amount of load given to the green pellet until fracture was developed was noted as GCS of the pellet. The arithmetic mean of the GCS values of ten pellets of each batch was taken as GCS of that particular batch Nayak[24]. Then the green pellets were subjected to heating in an oven at 100 °C for two hours to remove the moisture content Nayak[25].

2.2.4 The charge mix

Three sets of recipes [O1, O2 & O3] having 98%, 97.75% and 97.5% of < 150 µm size ochre sample with a fixed quantity of limestone [0.5%] and coke fines [0.5%] but variable quantity of bentonite [0.5%, 0.75% and 1% respectively] were blended properly in a high intensity mixer to prepare the pellet mix for pelletization Nayak[25].

Limestone was used to provide CaO to form calcium containing slag phases that improves slag bonding and pellet properties. Coke fines are used as a reducing agent, for internal heat generation and uniform heating, stabilisation of the temperature, improvement in pellet strength and optimisation of porosity, whereas bentonite, act as a binder, increases the elasticity and plasticity of pellets resulting in enhanced properties. The recipes prepared for the pelletization process is given in Table 2.

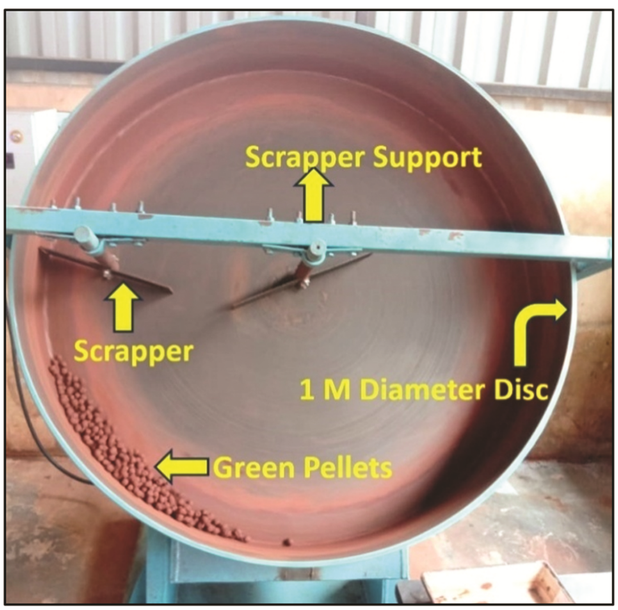

Fig. 2 — Picture of laboratory scale disc pelletizer used in the study [Disc Diameter- 1m; Disc Inclination- 47°; Disc Rotation- 20 RPM].

2.2.5 Induration of green pellets

The green pellets, produced after pelletization, needs to be indurated in a muffle furnace for a specific residence time to ensure proper oxidation of the constituent phases of pellets and the strength. The green pellets obtained from the three recipes (O1, O2 & O3) were indurated for a residence time of 10 minutes at three different temperatures such as 1100, 1200, and 1300 °C under air atmosphere to know the effect of induration temperature and residence time on pellet characteristics. The indurate pellets were cooled in the furnace to 600 °C followed by air cooling to room temperature before the product evaluation Nayak[25].

2.2.6 Product evaluation

2.2.6.1 Cold Crushing Strength (CCS)

In order to evaluate the quality of indurate pellets two tests such as: i) Cold Crushing Strength (CCS) and ii) Porosity need to be determined. The CCS refers to the amount of load required to break the indurated pellets. It is calculated according to ISO 4700:2007 by using a universal tensile testing system (Shanta Engineering, Kolkata) available at CSIR-IMMT. The procedure of calculation of CCS is as that of GCS (Green Compressive Strength). The arithmetic mean of CCS values of 20 pellets was taken as CCS of that particular batch Nayak[25], Sahu[26].

2.2.6.2 Porosity

The porosity of the indurated pellets was calculated using hot water boiling method according to ASTM C20 Nayak[24,25], Sahu[26].

3 Results and Discussion

3.1 Feed characterization

3.1.1 Mineralogical characteristics

The XRD pattern of two random ochre sample is presented in the Fig. 3. As one can see, the major mineral phases are hematite and goethite. The mineral assemblage identified through the X-ray diffraction was confirmed under optical reflected light microscope. The optical micrographs are presented in Fig 4. The goethite invariably shows a colloform texture Fig. 4(a). Tiny micro-platy hematite grains are

Table 2 — Recipes designed for the pelletization process

Recipes	Size, µm	Iron Ore (%)	Bentonite (%)	Limestone (%)	Coke Fines (%)
O1	-150	98	0.5	0.5	1
O2	-150	97.75	0.75	0.5	1
O3	-150	97.5	1.0	0.5	1

often seen with goethite and limonite Fig. 4(b). Occasionally, thin parallel lamella of hematite is seen in contact with lenticular goethite Fig. 4(c). Hematite sometimes shows reticular venation appearing like a mesh, filled with limonitic matrix (Fig. 4(d). This mineral assemblage in ochre plays a critical role in the pelletization process.

3.1.2 Chemical characteristics

The chemical composition of feed ochre sample along with the other charge mix: such as bentonite, lime and coke fines are given in Table 3 and Table 4. It shows that the Fe content in ochre is around 60%. The higher LOI (~10%) confirms the dominance of goethite over hematite. The Al_2O_3 and SiO_2 together

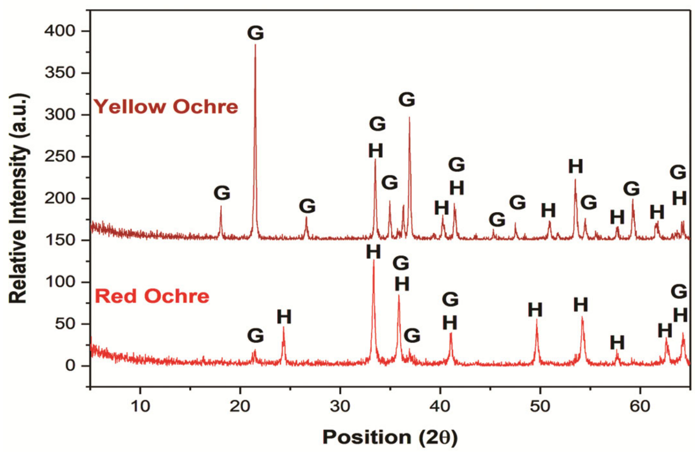

Fig. 3 — XRD pattern of Ochre sample (H-Hematite, G-Goethite).

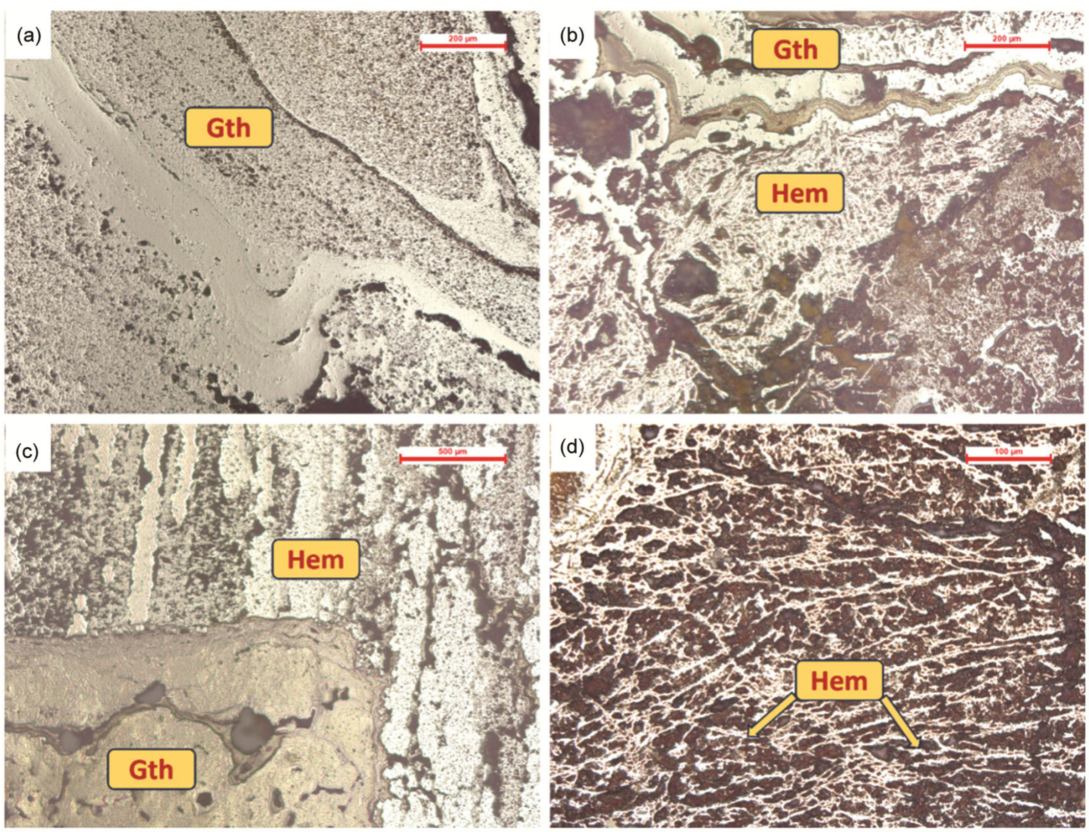

Fig. 4 — Photo micrographs of ochre sample under reflected light, a) Goethite showing a colloform texture, b) Micro-platy hematite grains dispersed within goethite and limonite, c) Hematite lamellae in contact with a colloform goethite, & d) Hematite showing reticular venation with limonitic matrix.

Table 3 — Chemical composition (in wt%) of the raw materials used in pelletization process

Sample	Fe	Al_2O_3	SiO_2	CaO	MgO	P_2O_5	MnO	TiO_2	Na_2O	LOI
Ochre	59.64	1.53	2.51	0.3	0.069	0.321	0.1	0.32	-	10.29
Bentonite	9.7	20.4		1.5	0.7	-	-	-	1.5	8.6
Lime	0.1	0.1	0.2	72.0	53.2	-	-	-	-	27.3

Table 4 — Proximate analyses of coke fines (in wt%) used for pelletization

Constituents	Moisture	Carbon	Volatile Matter	Ash
Coke Fines	3.0	81.1	3.3	13.6

Table 5 — Physical properties of green pellets

Properties	Recommended	Green Pellets		
		O1	O2	O3
Moisture (%)	8-10	11	12	10
Avg. Drop No.	>6.0	29	31	33
GCS (kg/pellet)	>1	2.93	3.18	3.27

contributes ~4% and the other compounds like CaO, MgO, SO_3, P_2O_5, MnO, are present in minor amount.

The compositional result of ochre indicates that it could be a suitable material for pelletization and the product quality can be improved by reducing the high LOI values.

3.2 Product Characterization

3.2.1 Characteristics of Green Pellet

During pelletization, good quality of green pellets should be produced which can bear the impact and pressure during transportation to the furnace. So, a standard quality is fixed for the green pellets. The recommended values of moisture (8-10%), average drop no. (> 6.0) and GCS (kg/pellet, > 1) are given in the Table 5.

The results show that the moisture content varies between 10% - 12% in all the sample irrespective of binder content. The GCS increases from 2.93 to 3.27 when the bentonite content increases from 0.5 to 1%.

3.2.2 Characteristics of indurate pellet

The characterization studies of indurated pellets are shown in Fig. 5. The indurate pellets are morphologically relatively smaller in size (~12 mm) than green pellets (14-15mm), may be because of its shrinking during indurations process (Fig. 5 (a&b).

Under optical microscope, though the grains in the pellets look more or less similar in size (Fig. 5 (c&d). under electron microscope, at higher magnification, an enlargement in grain size and marginal refinement is observed (Fig.5 (e&f). Two major phases such as hematite and fayalite with minor calcium ferrite are recorded from XRD analysis at all the temperature of indurations (Fig. 6), though minor increase in their quantity is noticed with rise of temperature from their peak intensity.

During induration all the goethite phases are converted to hematite which is the major mineral phase. With rise in induration temperature from 1100 °C to 1300 °C, the hematite concentration gradually increases as shown in Fig. 6. Along with hematite, other minerals like fayalite and calcium ferrite are also formed providing adequate strength to the pellets. The presence of higher amount of hematite phases suggests that the desired strength have been achieved by the pellets

The results of porosity (in %) and CCS (in kg/pellet) with variable bentonite content and indurations temperature are given in Table 6. One can observe from the table that as the bentonite content increases from 0.5% to 1% and the temperature raises from 1100 °C to 1300 °C the CCS value rises in each batch, while the porosity decreases. When the bentonite content is 1%, and the indurations temperature is 1300 °C, the CCS value is highest i.e. 255 kg/pellet and the porosity value is minimum (21.8%). In contrast, at lower bentonite content (0.5%) and low temperature of indurations (1100 °C), the CCS value decreases to ~98 kg/pellet having 42% porosity and the products are not acceptable / suitable to be used as blast furnace feed. A graphical presentation on the effect of binder and induration temperature on porosity and CCS is shown in Fig. 7.

3.3 Discussion

When the ochre in < 150 µm size from the mine profile (appearing both in yellow and red colour) of Daitari iron ore deposit is pelletised in a disc pelletiser along with appropriate charge mix, small green pellets are formed. Samples in three sets (O1, O2 & O3) were taken with variable bentonite [0.5, 0.75 and 1%] but fixed lime and coke content. The physical properties of green pellets such as moisture content (in%), average drop number and GCS (kg/pellet) were determined. All the three sets were found to be within recommended value.

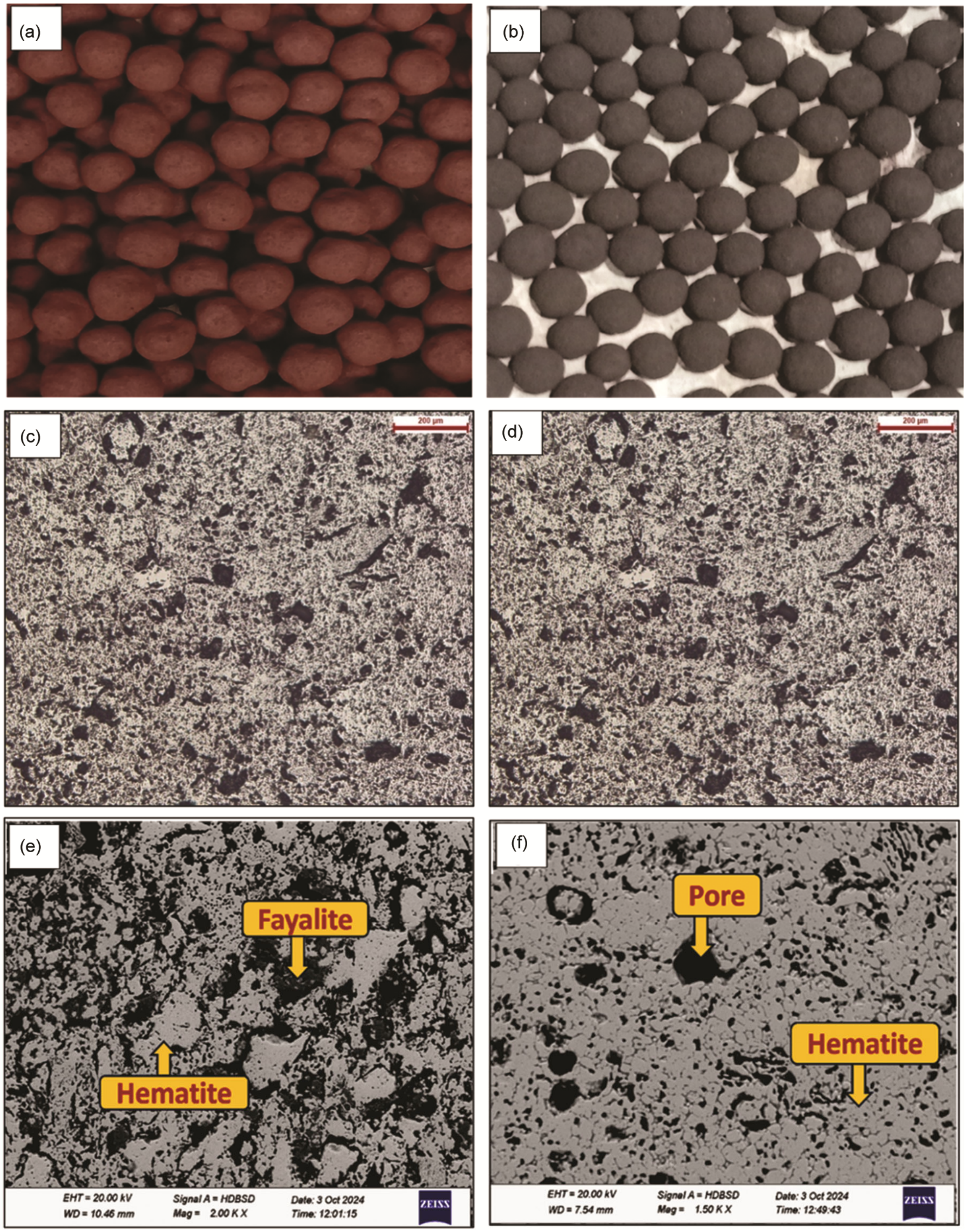

Fig. 5 — Green & Indurate pellets; and microscopic characteristics of indurate pellets a) Green pellets, b) Indurated pellets, (c&d). Optical micrographs of indurate pellets at different magnification, and (e&f). SEM images of indurated pellets at different magnification at 1100 °C and 1300 °C.

All the three sets of green pellets were indurated in a muffle furnace at three different temperatures such as 1100, 1200 and 1300 °C for a residence time of 10 minutes each.

During induration all the goethite phases present in ochre sample are converted to hematite and thereby add to the hematite content already available. Along with hematite, molten slag phase led to development

Table 6 — Physical properties of indurated pellets

Recipe Code	Bentonite (%)	Induration Temperature (^{O}C)					
		1100		1200		1300	
		Porosity (%)	CCS (kg/pellet)	Porosity (%)	CCS (kg/pellet)	Porosity (%)	CCS (kg/pellet)
O1	0.5	42	98	34.1	150.1	25.1	225
O2	0.75	41.5	105	33.6	180	24	245.3
O3	1.0	40.7	110	31.53	182.6	21.8	255.2

Table 7 — Chemical analysis results of indurated pellets (in wt %)

Induration Temp (^{o}C)	Bentonite (%)	Fe	Fe_2O_3	Al_2O_3	SiO_2	CaO	P_2O_5	MnO	TiO_2
1100	1	65.8	94.80	1.9	1.5	0.74	0.3	0.16	0.4
1200	1	66.5	95.09	1.8	1.4	0.49	0.4	0.10	0.43
1300	1	67.77	96.91	1.83	1.37	0.7	0.23	0.17	0.35

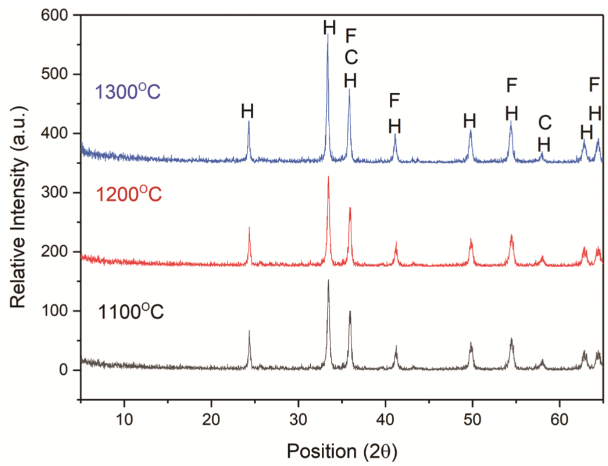

Fig. 6 — XRD pattern of Indurate Pellet at different temperature H-Hematite; F-Fayalite, C-Calcium Ferrite.

of recrystallized iron silicate (fayalite) and calcium iron oxide phase (calcium ferrite), which are responsible for strengthening of pellets Pal[27], Gan[28]. As the induration temperature rises from 1100 °C to 1200 °C, the hematite concentration gradually increases and when induration temperature increases from 1200 °C to 1300 °C, the fayalite and calcium ferrite content also increases Devasahayam[29].

These indurate pellets were evaluated in respect of their CCS (kg/pellet) and porosity (%) value. It is observed that the pellets having 1% bentonite, indurated at 1300 °C gave the best result and would be suitable for its use in blast furnace for iron making. At this temperature (1300 °C) the pellets are less porous and much superior than pellets indurated at other two temperatures. It indicates that higher the induration temperature, better the crystallisation of hematite and superior the pellet quality Jang[30].

Chemical analysis results of the indurate pellets prepared from feed size of -150 µm size ochre sample

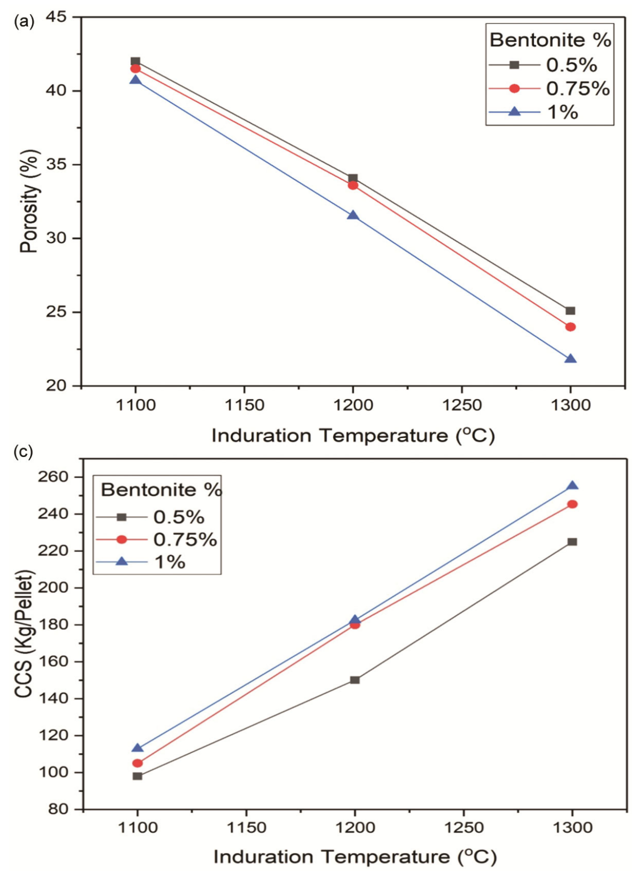

Fig. 7 — Plot showing the effect of binder and induration temperature on porosity (top) and CCS (bottom) of pellets.

with varying bentonite content (0.5%, 0.75% and 1%) and indurated at 1100, 1200 and 1300 °C are presented in the Table 7. Pellets indurated at 1300 °C shows a rise in Fe concentration from ~60% to ~67%, with combined Al_2O_3 + SiO_2 content of ~3.2%, making a suitable material for iron and steel making. Other constituents such as CaO, P_2O_5, MnO and TiO_2 show minor changes from their respective feed.

4 Conclusions

From the ongoing discussion the followings conclusions are brought out.

i) The Ochre present in different iron ore deposit in India, Daitari iron ore deposit in particular, though have appreciable Fe content, can't be directly used in blast furnace for iron making due to its powdery nature. This article discusses the method to convert these fines into lumpy form using pelletization technique.

ii) The run-of -mine ochre sample in Daitari deposit is composed of ~60% of Fe, ~4% of combined Al_2O_3 + SiO_2 and ~10% LOI. The mineralogical study shows it to have major hematite and goethite with minor limonite and clay.

iii) Pellets were prepared in a disc pelletizer from three different sets of ocher sample with fixed limestone, coke but variable bentonite content.

iv) Optimization of different parameters were undertaken to meet the required standard in terms of GCS, CCS, porosity etc.

v) The physical properties of green pellets such as moisture content, average drop number and Green Compressive Strength (GCS) in all the sets were found to be within the recommended value.

vi) These three sets of green pellets were indurated in a muffle furnace at three different temperatures, viz. 1100, 1200 and 1300 ^{o}C for 10 minutes residence time in each case. Pellets prepared with 1% bentonite content indurated at 1300 ^{o}C gave desired value of 22% porosity and 255 CCS (kg/pellet).

vii) The characterization of the indurated pellets under optimal conditions indicates that the recrystallization of hematite during induration process provides optimum strength to the pellets through diffusion bonding formation. The formation of fayalite, calcium ferrite also supports stability to the pellet by slag banding.

viii) Pellets indurated at 1300 ^{o}C shows a rise in Fe concentration from ~60% to ~67%, with combined Al_2O_3 + SiO_2 content of ~3.2%, making a suitable material for sustainable iron making.

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